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VOLUMETRIC DETERMINATION OF SMALL AMOUNTS OF
REDUCING SUGARS BY A MODIFIED LANE-EYNON PROCEDURE X

by C. A. Fort
Agricultural Chemical Research Division
Bureau of Agricultural and Industrial Chemistry
United States Department of Agriculture
New Orleans 19, Louisiana

Because sugar beets are low in reducing substances, it has been necessary to modify the usual Lane-Eynon procedure for their determination. Briefly, the modification is a supplemental titration, by a standardized invert sugar solution, of the residual strength remaining in a 10-ml. portion of Fehling solution after it has reacted with a measured volume of the unknown.

Reagents

Sexhlet modification of Fehling solution

Prepare by mixing equal volumes of solutions

(a) and (b) immediately before use.

(a) Copper sulphate solution: 69.278 grams of

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ dissolved in water and made up to a volume of 1 liter in volumetric flask. Crystals used should not show efflorescence.

(b) Alkaline solution: 346 grams of dissolved sodium potassium tartrate (Rochelle salt) plus 100 grams of sodium hydroxide and when cooled to room temperature made up to 1 liter.

Methylene blue solution: 1 gram dissolved in water and made up to 100 ml.

Standardized solution of invert sugar: preferred concentration is 210 mg. invert sugar per 100 ml. volume.

Preparation: Into a 100-ml. volumetric flask put 20 grams of dry sucrose and add about 50 ml. of water. When in solution add 2 or 3 drops of glacial acetic acid and 10 ml. of invertase (regular 0.1 k activity).^{*} Let stand overnight at room temperature (70-80° F.). When inverted make up to the 100 ml. volume and store in refrigerator. This concentrated solution normally keeps for several weeks. When used the concentrated invert is diluted, 10 ml. to 1 liter. (warm concentrate to room temperature before pipetting.) Even this dilute invert will keep 2 or 3 days but it is best to make a dilution each day. One milliliter of the dilute contains 2.1 mg. of invert.

Standardization: To check the quality of the reagents a direct titration of the dilute standard invert may be made by the regular Lane-Eynon procedure. For example, to 10 ml. of mixed Fehling in a 300-ml. Erlenmeyer flask add roughly 24 ml. of the standard invert from a burette (burette should have an offset delivery tip to reduce heating of the burette while titrating). Bring to a boil in about 2 minutes and let boil for 1-1/2 minutes; then add 3 drops of methylene blue and complete the titration slowly dropwise. The total boiling time should be between 2-1/2 and 3 minutes. The final titre should be ^{24.4}~~24~~ ml. if reagents are perfect. Variations of plus or minus 0.1 ml. are unimportant.

Standardization by the above procedure establishes the concentration of the invert sugar. It is desirable to check the quality of the

^{*}In lieu of preparing invertase or purchasing invertase solutions of unknown concentrations, it is simpler to obtain "Invertase Scales" (blue label) from Wallerstein Laboratories, 180 Madison Ave., New York 16, N. Y. A water solution of 0.1 gram of this product per 100 ml. volume will suffice for the inversion. It is not the policy of the Department to recommend the products of one company over those of any others engaged in the same business. This name is furnished merely for your convenience and information.

reagents in this manner once each day. This will primarily check the concentrations of the new dilution of invert sugar which is subject to some variations due to pipetting the fairly viscous concentrated invert solution. If the deviation from the 24.4 ml. titre value is over 0.2 ml., it will be possible to make adjustment for this later in the procedure.

Control Titration

The object of the control titration is to compensate for the sucrose and the dilution incurred when testing the unknown. It needs to be established only once for each type of product that is to be analyzed..

To a 10 ml. portion of mixed Fehling add 25 ml. of a sucrose solution whose concentration corresponds to the approximate average concentration of the solution of the product being analyzed. With half-normal beet diffusates (i.e., a half-normal weight per 100 ml. or a normal weight made up to 200 ml.), the usual sucrose concentration is roughly 2 grams per 100 ml. volume. The normal variation in sucrose percent beets need not be considered; however, in extreme cases where the sucrose is below 13% or over 18% it would be best to adjust the concentration of the sucrose solution used in the control titration. For molasses or other sugar products the approximate mean concentration of sucrose per 100 ml. of the test solution would be the concentration of sucrose used in the control titration. With invert sugars only, as after inversion of the product to determine true sucrose, then 25 ml. of distilled water is used for the control titration.

After adding the 25 ml. of the proper sucrose solution, add about 24 ml. of standard invert and make a titration as in the standardization. This titration will be about as follows:

For 25 ml. water only	25.3 ml.
25 ml. of 0.5 g. sucrose per 100 ml.	25.0 ml.
25 ml. of 1.0 g. sucrose per 100 ml.	24.8 ml.
25 ml. of 2.0 g. sucrose per 100 ml.	24.6 ml. (beet diffusates)
25 ml. of 4.0 g. sucrose per 100 ml.	24.1 ml.

These values are merely illustrative; the analysts should use values which they obtain for themselves.

The control titration should be repeated several times until it is certain that the value is established. When the titre of the standard invert differs from the value of 24.4 ml. mentioned under standardization, the control titration value may be adjusted arithmetically. For example, if the standardization titre were 24.7 ml., then 0.3 ml. would be added to the established control titration value.

To emphasize, the control titration need only be established once for a particular product and concentration of sucrose; but a standardization of the invert solution should be made each day, which usually involves merely one titration. This latter value may, when necessary, be used to correct the control titration figure.

Determination

To 10 ml. of mixed Fehling add 25 ml. of deloaded diffusate, or other clarified and deloaded product, and make an addition of the standard invert from the burette. The amount of this initial addition of the standard invert can be 20 ml. with diffusates of sound beets. (If this proves to be too much, the test must be repeated until the amount added is insufficient to complete the Fehling reaction. If the 25 ml. of the solution being tested is by itself an excess, then the usual direct titration of the Lane-Eynon procedure is used.). After the mixture has boiled 1-1/2 minutes, the standard

invert is added rapidly, though dropwise, to obtain a trial end point. The test is then repeated making the preliminary addition of invert about 0.5 ml. less than the trial titre, and the titration completed in the regular manner adding the final amounts of invert very slowly. If the final titre obtained is more than 1 ml. above the amount of the initial invert addition, the determination should be repeated again.

Calculation

Subtract the titre of the unknown from the control titre. This difference represents the titre of the 25 ml. of the sample in terms of the standard invert. Hence, the difference in titre is multiplied by the 0.0021 g. concentration per ml. of invert and divided by the weight of product in the 25 ml. aliquot used.

Example (based on half-normal weight cossette diffusate):

Control titre,	24.7 ml.
Sample titre,	23.0 ml.
Difference,	1.7 ml.

$$\frac{1.7 \times 0.0021 \times 100}{3.25} = \frac{0.357}{3.25} = \underline{0.11\%}$$

